

# memorandum

Carlsbad Field Office  
Carlsbad, New Mexico 88221

DATE: July 29, 2003

REPLY TO  
ATTN OF: CBFO:QA:MLC:GS:03-2513:UFC 2300.00

SUBJECT: Audit (A-03-24) of Los Alamos National Laboratory New Headspace Gas Unit and Manual Headspace Gas Sampling

TO: James Nunz, LASO

Attached is Audit Report A-03-24 of the Headspace Gas Sampling and Analysis using the Entech/Agilent system at Los Alamos National Laboratory. The audit was conducted July 8-10, 2003. If you have any questions or comments concerning the audit, please contact me at (505) 234-7442.

M. Lea Chism  
Quality Assurance Specialist



## Attachment

cc: w/attachment  
K. Watson, CBFO \*ED  
R. Erickson, LASO \*ED  
G. Rodriguez, LASO \*ED  
D. Newell, LASO \*ED  
S. Wander, LANL \*ED  
P. Lindahl, LANL \*ED  
M. Eagle, EPA \*ED  
E. Feltcorn, EPA \*ED  
S. Zappe, NMED \*ED  
S. Holmes, NMED \*ED  
B. Walker, EEG \*ED  
D. Winters, DNFSB \*ED  
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U.S. DEPARTMENT OF ENERGY  
CARLSBAD FIELD OFFICE

DRAFT AUDIT REPORT

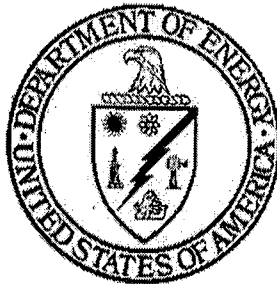
OF THE

LOS ALAMOS NATIONAL LABORATORY  
LOS ALAMOS, NEW MEXICO

WASTE CHARACTERIZATION ACTIVITIES  
Headspace Gas Sampling and Analysis

AUDIT NUMBER A-03-24

July 8 – 10, 2003



Prepared By: Thomas E. Putnam  
Thomas Putnam  
Audit Team Leader

Date: 7-29-03

Approved By: Ava L. Holland  
Ava L. Holland  
CBFO QA Manager

Date: 7/30/03

## 1.0 EXECUTIVE SUMMARY

Carlsbad Field Office (CBFO) Audit A-03-24 was conducted to evaluate the adequacy, implementation, and effectiveness of obtaining headspace gas (HSG) sampling, analysis and associated activities utilizing the Entech/Agilent system.

The audit was conducted at the Los Alamos National Laboratory (LANL) in Los Alamos, New Mexico, July 8-10, 2003. The audit team concluded that the LANL process for obtaining manual HSG samples was adequate relative to the flow-down of requirements from the CBFO Quality Assurance Program Document (QAPD) and the Hazardous Waste Facility Permit (HWFP). The audit team also concluded that the LANL technical processes were satisfactorily implemented and effective.

The audit team identified four isolated deficiencies requiring only remedial corrective actions that were corrected during the audit (CDA). Six recommendations were offered for management consideration.

## 2.0 SCOPE

The scope of the audit was to evaluate LANL processes for adequacy, implementation, and effectiveness in performing sampling, analysis, canister cleaning, sample port installation, helium leak testing, batch data report generation, and the review of the batch data associated with the Entech/Agilent system. Compliance with the WIPP HWFP Waste Analysis Plan (WAP) and selected portions of the CBFO QAPD was also evaluated.

The following Quality Assurance (QA) elements were evaluated:

- Personnel Qualification and Training
- Measuring and Test Equipment
- Sample Control

The following characterization technical elements were evaluated:

- HSG Sampling and Analysis
- Canister Cleaning
- Sample Port Installation
- Helium Leak Testing
- Generation Data Review
- Project-level Data Verification and Validation

The evaluation of LANL documents was based on the current revisions of the following documents:

- *CBFO Quality Assurance Program Document, CAO-94-1012*
- *WIPP Hazardous Waste Facility Permit*
- Related LANL technical and QA implementing procedures

### **3.0 AUDIT TEAM AND OBSERVERS**

#### **AUDITORS/TECHNICAL SPECIALISTS**

Thomas Putnam	Audit Team Leader, CTAC
Dorothy Gill	Technical Specialist, CTAC

#### **INSPECTORS/OBSERVERS**

Steve Holmes	New Mexico Environment Department (NMED)
Scott Webb	Environmental Evaluation Group (EEG)

### **4.0 AUDIT PARTICIPANTS**

LANL personnel participating in this audit process are identified in Attachment 1. A pre-audit meeting was held in the Oppenheimer Building on July 8, 2003. A daily meeting was held with LANL management and staff to discuss issues and potential deficiencies. The audit was concluded with a post-audit meeting held in the Oppenheimer Building on July 10, 2003.

### **5.0 SUMMARY OF AUDIT RESULTS**

#### **5.1 Program Adequacy, Implementation, and Effectiveness**

The audit team concluded that the LANL technical and QA processes/procedures were adequate, relative to the flow-down of requirements from the CBFO QAPD and the HWFP. The audit team also concluded the LANL technical processes were satisfactorily implemented and effective.

The audit team concluded that the defined LANL QA program elements reviewed were adequate and satisfactorily implemented in accordance with the LANL Quality Assurance Program Manual (QAMP), the LANL Quality Assurance Project Plan (QAPJP), and LANL implementing procedures for the areas evaluated. The LANL QA program in those areas was also determined to be effective. For details of CARs, CDAs, observations, recommendations, and exemplary practices, see Section 6.

A summary table of audit results for each of the QA program elements and the technical processes is provided in Attachment 2. Audit activities, including the specific objective evidence reviewed, are described below. A list of procedures evaluated during the audit is included in Attachment 3.

#### **5.2 Quality Assurance Activities**

Details of the objective evidence reviewed in the QA areas are contained in the audit records. The QA activities evaluated were determined to be adequate, satisfactorily implemented, and effective.

### 5.3 Technical Activities

Evaluations of applicable LANL technical activities are summarized below.

#### 5.3.1 Headspace Gas Sampling and Analysis (Entech/Agilent System)

Direct canister headspace gas sampling and associated activities were reviewed during the audit. The activities audited were documented in the following procedures:

- TWCP-DTP-1.2-069, *Installation of the NucFil HGAS Sample Port*
- TWCP-DTP-1.2-070, *Canister Cleaning Using Entech 3100 Canister Cleaning System*
- TWCP-DTP-1.2-071, *Manual Headspace Gas Sampling of LANL TRU Waste Containers*
- TWCP-DTP-1.2-072, *TRU Waste Container HGAS Analysis (Entech/Agilent)*
- TWCP-DTP-0.0-078, *Headspace GAS Sampling and Analysis Batch Data Reports Preparation (Entech/Agilent)*
- TWCP-DTP-0.0-079, *Entech Canister Gauge Leak Test*

Headspace gas sampling and analysis operations were the subject of the audit activities. The activities reviewed and inspected were: sample port installation, direct canister sampling, GC/MS analysis, canister cleaning, canister gauge leak test, and generation and review of batch data reports. The audit included observation of sample port installation, headspace gas sampling (for the batch sampled on 7/8/03), and demonstration of the Entech canister cleaning system. Operation of the Entech autosampler, used to introduce headspace gas samples to the analytical instrument (GC/MS), and the processing of analytical data were also observed. Additionally, documents and records were inspected, various personnel interviewed, and batch data reports reviewed.

All areas inspected were well organized, and the samplers, technicians, analysts and supervisors knowledgeable with regard to their individual duties. The operational systems implemented by the headspace gas team were technically sound, suitable for use, and well documented.

#### 5.3.2 Project Level Data Validation and Verification

Project level data validation and verification activities associated with the Entech/Agilent headspace gas unit were reviewed during the audit. This activity is documented in LANL procedure QP-1.1-010, *Project Level Data Validation and Verification*.

Various documents and records were inspected, reviewers interviewed and data packages reviewed. The reviewers were knowledgeable with regard to their duties, documents and records complete, and the data packages provided the necessary acceptance information to ship TRU waste.

## **6.0 CORRECTIVE ACTION REPORTS (CARs), CORRECTED DURING THE AUDIT (CDAs) OBSERVATIONS, RECOMMENDATIONS AND EXEMPLARY PRACTICES**

During the audit, the audit team may identify conditions adverse to quality (CAQ) and document such condition(s) on corrective action reports (CARs).

**Condition Adverse to Quality (CAQ)** – An all-inclusive term used in reference to any of the following: failures, malfunctions, deficiencies, defective items, nonconformances, and technical inadequacies. A significant condition adverse to quality is one that, if uncorrected, could have a serious effect on safety, operability, waste isolation, TRU waste site certification, regulatory compliance demonstration, or effective implementation of the QA program.

**Significant Condition Adverse to Quality** – A condition which, if uncorrected, could have a serious effect on safety, operability, waste confinement, TRU waste site certification, compliance demonstration, or the effective implementation of the QA program.

### **6.1 Corrective Action Reports (CARs)**

There were no CARs identified by the audit team during this audit.

### **6.2 Corrected During the Audit**

During the audit, the audit team may identify conditions adverse to quality (CAQ). The audit team members and the audit team leader (ATL) evaluate the CAQs to determine if they require a CAR. Once a determination is made that the CAQ does not require a CAR, the audit team members, in conjunction with the ATL, determine if the CAQ is an isolated case requiring only remedial action and, therefore, can be corrected during the audit (CDA). Upon determination that the CAQ is isolated, the audit team members, in conjunction with the ATL, evaluate/verify any objective evidence/actions submitted or taken by the audited organization and determine if the condition was corrected in an acceptable manner. Once it has been determined that the CAQ has been acceptably corrected, the ATL categorizes the condition as CDA.

**Corrected During the Audit (CDA)** – Isolated deficiencies that do not require a root cause determination or actions to preclude recurrence, and correction of the deficiency can be verified prior to the end of the audit. (Examples: one or two minor changes required to correct a procedure (isolated); one or two forms not signed or not dated (isolated); one or two individuals that have not completed a reading assignment.)

Four isolated deficiencies, requiring remedial action only, were identified during the audit and reported as CDAs. They were corrected and verified before the completion of the audit.

#### **6.2.1 CDA 1**

Not following procedure TWCP-DTP-1.2-069, Installation of the Nucfil HGAS Sample Port. During the checking of the drum thickness it was noted that the lid measured

0.0529 and the procedure required that the pounds per square inch (psi) be set at 90 to install the sample port. However the operator stated that he rounds up when the reading is boarder line, in this case he rounded up to 0.0530 and the procedure requires a psi of 110. The procedure did not allow for rounding of the measured reading.

An interim change request was implemented that added a sentence and bullets to section 7.4.13. "If the digital ultrasonic micrometer is a model that reads out more than three figures after the decimal place for the drum thickness listed in table 1, round the number in accordance with established rounding rules.

- If the digit 5,6,7,8, or 9 is dropped, increase the preceding unit by 1 unit
- If the digit 0,1,2,3, or 4 is dropped, do not alter the preceding digit"

#### **6.2.2 CDA 2**

Procedure TWCP-DTP-0.0-071, Manual Headspace Gas Sampling of LANL TRU Waste Containers does not contain information on how the canisters are packaged for transportation to the laboratory. Also the procedure does not specify that the chain of custody form (COC) for canisters sampled is completed and placed in the cooler to be taken to the laboratory.

An interim change request was implemented that added a new section 8.7.5 "Samples are packed in appropriately cushioned and secured packaging so as to avoid canister damage during transport to the laboratory. For each sample batch, the COC Form will be packaged together with samples."

#### **6.2.3 CDA 3**

The temperature of the refrigerator being used to store VOC liquid standards was measured at 1.3°Centigrade (C) on 7/1/03 and 0.8° C on 7/9/03. Laboratory personnel stated that it should be stored at 4°C. Also the storage requirement for liquid VOC standards is not specified in procedure DTP-0.0-072.

An interim change request was implemented that added a the second sentence to the bullet beginning "Custom liquid standard-" "Standards will be stored at 4°C or in accordance with manufacturer's specifications."

#### **6.2.4 CDA 4**

Procedure DTP-0.0-072 does not include all of the options used to select 4-bromofluorbenzene (BFB) scan(s) used for tuning the mass spectrometer. SW-846, Method 8260B Section 7.3.1.1 requires the laboratory to have a documented approach to scan selection.

An interim change request was implemented that replaced the Note at the end of section 7.4.5.2.c with the following text. "Three methods of finding passing BFB may be used. First, check the spectrum at the apex of the BFB peak. Second, average three

scans, the peak apex scan, and the scans immediately preceding and following the apex. Finally, average scans through the peak. These three methods may be used with or without background subtractions. For background subtraction use a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak."

### **6.3 Observations**

There were no observations identified by the audit team during the audit.

### **6.4 Recommendations**

The following recommendations are provided for management consideration.

#### **6.4.1 Recommendation 1**

After the sampling event is completed, place a suitable trip blank in the cooler with the samples.

#### **6.4.2 Recommendation 2**

The result sheet for the continuing calibration does not show the %D for hydrogen and methane. Hence it is not possible to determine from the sheet if the continuing calibration requirements were met. It is recommended that the sheet be revised to include this information.

#### **6.4.3 Recommendation 3**

SW 846 Method 8260B, section 5.14 states that "all standards in methanol be stored at -10°C or less". The liquid VOC standards used by the laboratory are mixtures of pure compounds and are not dissolved in methanol. However, the standards contain methanol and it is recommended that appropriateness of storing these standards at 4°C be investigated.

#### **6.4.4 Recommendation 4**

The results for hydrogen are reported to either two or 3 decimal places (e.g., batch LA03-HGAS/LA-001 hydrogen for drum 959196 was reported to 2 decimal places and that for drum 959150 it was reported to three decimal places). A consistent reporting format should be applied.



#### **6.4.5 Recommendation 5**

The chromatograms for the quantitation reports have over-written analyte names that make them unreadable. It is recommended that the format be changed to allow all analyte names to be readable.

#### **6.4.6 Recommendation 6**

The Note on the "Gas sample Chain of Custody" form from DTP-0.0-071 uses the words "sign and initial". However the samplers printed their names and initials. It is recommended that the form be changed to allow the printing of names or the samplers instructed to sign their names.

### **7.0 LIST OF ATTACHMENTS**

- Attachment 1: Personnel Contacted During the Audit
- Attachment 2: Summary Table of Audit Results
- Attachment 3: List of Procedures Audited

**PERSONNEL CONTACTED DURING AUDIT A-03-07**

NAME	ORG/TITLE	PREAUDIT MEETING	CONTACTED DURING AUDIT	POST AUDIT MEETING
Anghel, Ioana	LANL/RRES-CH		X	X
Ankon, James	LANL/RRES-WDS		X	
Bailey, James	LANL/RRES-CE			X
Burt, Jean	LANL/RRES-QA		X	
Coriz, Suzanne	LANL/RRES-CH			X
Del Signore, J.C.	LANL/Project Manager	X		X
Fernandez, Ruby Ann	LANL/RRES-CE	X		X
Garcia, Mary Ann	LANL/RRES-CE	X		X
Gibson, Yvonne	LANL/RRES-CE	X		
Hardesty, Bill	LANL/HSG	X	X	X
Hartwell, Ware	LANL/RRES-QAT			X
Huchton, Judith	LANL/RRES-CE	X		X
Humphrey, Betty	LANL/SPM	X	X	X
Lindahl, Peter	LANL/SPQAO	X		X
Lopez, Joshua	LANL/RRES-WDS		X	
Marczak, Stanislaw	LANL/RRES-CH	X	X	X
Martin, Beverly	LANL/RRES-WD			X
Miller, Scott	LANL/RRES-CH	X		
Mullen, Richard	LANL/RRES-WDS		X	
Polley, Mark	RRES-AT/TCO			X
Powell, Mark	LANL/RRES-QAT		X	X
Newell, Dorothy	LASO-OPL			X
Nunz, James	LASO-OFO			X

NAME	ORG/TITLE	PREAUDIT MEETING	CONTACTED DURING AUDIT	POST AUDIT MEETING
Riggs, Matt	LANL/RRES-CE			X
Romero, Eric	LANL/RRES-WDS		X	
Saunders, Lori	LANL/QA			X
Sullivan, Jeri	LANL/RRES-CH		X	X
Uecker, Barbara	LANL/RRES-OEIM	X		X
Velasquez, Carmen	LANL/RRES-CE	X		X
Vigil, Chris	LANL/RRES-WDS		X	
Wander, Sandy	LANL/RRES-CE	X		X

# Summary Table of Audit Results

Evaluation Area	Concern Classification			QA Evaluation		
	EP	CARs	CDAs	Obs	Rec	Effectiveness
Headspace Gas Sampling			2		4	E
Sample Control			2		2	E
Project Level V&V						E
Training						E
Measuring and Test Equipment						E
<b>TOTALS</b>			<b>4</b>		<b>6</b>	<b>E</b>

## Definitions

E = Effective  
S = Satisfactory  
U = Unsatisfactory  
I = Indeterminate  
A = Adequate  
M = Marginal

CDA = Corrected During Audit  
CAR = Corrective Action Report  
Obs = Observation  
Rec = Recommendation  
EP=Exemplary Practice

## PROCEDURES AUDITED DURING A-03-24

NUMBER	PROCEDURE NUMBER	TITLE
1.	QP-1.1-003	TWCP Training
2.	QP-1.1-010	Project Level Data Validation and Verification
3.	QP-1.1-018	Measuring and Test Equipment
4.	DTP-1.2-069	Installation of the NucFil HGAS Sample Port
5.	DTP-0.0-070	Canister Cleaning Using Entech 3100 Canister Cleaning System
6.	DTP-1.2-071	Manual Headspace Gas Sampling of LANL TRU Waste Containers
7.	DTP-1.2-072	TRU Waste Container HGAS Analysis (Entech/Agilent)
8.	DTP-0.0-078	Headspace Gas Sampling and Analysis Batch Data Reports Preparation (Entech/Agilent)
9.	DTP-0.0-079	Entech Canister Gauge Leak Test